

## Abstract for Hydrogen Workshop

### Determination of Hydrogen by Cold Neutron Prompt Gamma-ray Activation Analysis

Rick L. Paul, National Institute of Standards and Technology, Gaithersburg, MD 20899

The presence of even trace amounts of hydrogen affects the properties of many materials. However, few techniques yield reliable quantitative measurement of hydrogen at these levels. We have found prompt gamma-ray activation analysis (PGAA) to be useful for the measurement of hydrogen in a variety of materials. During sample irradiation, elemental nuclei undergo neutron capture and emit prompt gamma-rays upon de-excitation. The gamma-rays are then measured using a high purity germanium detector. Comparison with standards yields quantitative multielemental analysis. The measurement is nondestructive, chemically specific, matrix independent, and because both neutrons and gamma-rays are penetrating, the entire sample is analyzed. Furthermore, because the measurement arises from a nuclear reaction, the results are independent of the chemical form of the element being determined. The hydrogen peak at 2223 keV has few spectroscopic interferences.

An instrument for PGAA is operational in the cold neutron guide hall of the NIST Center for Neutron Research. Neutrons from the reactor core, reduced in energy by passage through a liquid hydrogen cold source, pass through a  $^{56}\text{Ni}$  coated guide to the PGAA station. The use of cold neutrons and guides results in better sensitivity and lower background. Samples may be irradiated in an evacuated sample chamber to reduce hydrogen background due to atmospheric water vapor. The detection limit for hydrogen in many materials is  $< 10$  mg/kg. The instrument has been used to measure hydrogen and other elements in a wide variety of materials. An area of particular concentration has been the measurement of hydrogen in metals. Titanium causes embrittlement in titanium alloys at mass fractions about  $\sim 125$  mg/kg. We have used PGAA to measure hydrogen in a titanium alloy jet engine compressor blades that failed during service. Mass fractions up to 750 mg/kg were determined, indicating that hydrogen embrittlement could not be ruled out as a cause of failure. We are currently using PGAA to certify mass fractions of Standard Reference Materials made by doping the titanium alloy with a known amount of hydrogen, which are being produced as calibration standards for industrial measurement of hydrogen by hot extraction. The first of these SRMs, certified at  $114 \pm 5$  mg/kg H has been produced, and production of two SRMs of bracketing concentrations is underway. NIST is also currently working with Jefferson Laboratory on the problem of measuring hydrogen in niobium being used in the construction of the accelerator for the Spallation Neutron Source. PGAA was used to measure hydrogen mass fractions of near 30 mg/kg in five samples niobium before and after chemical treatment. Elevated levels after treatment were found in only one sample.